

Microscopic Analysis of Carbon Nano Tubes based Metal Matrix Composite

V. J. Pillewan, D. N. Raut and K. N. Patil

Abstract—These days significant metal waste is generated during the machining operations such as; grinding, milling, shaping, forging etc. This waste management will pose a big challenge for the industry due to environmental litigation. Grinding industry, the waste contains organic and inorganic compounds, which will be difficult to reprocess. Current work focus to reuse and produce a value-added product from the grinding waste by Powder Injection Molding (PIM) approach. As the PIM product do not possess good mechanical properties, such as less yield strength, wear resistance, corrosion resistance, etc. To improve some of these properties, Carbon nanotubes (CNTs) are added. To observe the microstructure and porosity, the PIM products are tested with SEM analysis. It is observed that the products are porous in nature however it possesses good hardness. The hardness testing suggests that the PIM product will have higher hardness number with less porosity.

Index Terms—Metal Matrix Composites; Powder Injection Molding; Carbon Nanotubes; Grinding Waste.

I. INTRODUCTION

A metal matrix composite is prepared by dispersing a reinforcing material and fiber reinforced material. The reinforcement surface can be coated to prevent chemical reaction with the matrix. The reinforcement serves a structural task and used to change the physical properties such as wear resistance, friction coefficient or thermal conductivity. The reinforcement can be either form of discontinuous or continuous fibers. Discontinuous MMCs can be isotropic in nature. While, continuous reinforcement uses monofilament wires or fibers such as carbon fiber or silicon carbide fiber. The direction of the woven structure will result in an anisotropic structural form in which the alignment of material affects its strength. The first MMCs used were boron filament as reinforcement material[1]. The applications for MMC are driven by a lightweight, high-stiffness material and/or for elevated temperature resistance in structures. Typically MMC is widely used in aerospace industry. The specific modulus (elastic modulus divided by density) has been shown to be two to four times more to that of high-strength structural metals. The specific strength of MMC will be substantially higher than the parent metal.

Carbon nanotubes (CNTs) have found an increasing interest as a reinforcement material for iron/aluminum matrix due to their excellent mechanical properties such as extraordinary

stiffness and strength, which are unparalleled by any other material available today[2], [3], [4], [5], [6]. Thus, CNTs are extensively used for fiber refinement in MMC. The MMC prepared with CNT reinforced material found applications in the aerospace, automotive and sports industries where light weight combined with high stiffness and strength is desired. It has been studied widely the role of CNTs on the strengthening mechanism of the metal matrix. Apart from enhancement in elastic modulus, it has also been observed that CNTs obstruct dislocation movement. Thus, creating high dislocation density in the metal matrix composites prepared with the different materials such as Al[7], Mg [8], [9], [10], [11], Cu[12] and lead-free solder (Sn/Ag/Cu) alloy[13]. Recently, research on CNT reinforced metal-matrix composites have been increasing and the results show the possibility of both high strength structural and functional applications with high mechanical and thermal properties [14]. CNTs and graphene reinforced MMC decreases both coefficient of friction and wear rate as well as increases the tensile strength [15]. However, it is observed that the parentage amount of CNTs is required in large quantity. The reported literature suggested that the CNT yield is too low for the batch process[16]. The reported yield of CNTs ranges only a few milligrams to grams per batch[17]. Moreover, the carbon to CNTs conversion efficiencies is very poor thereby increasing the cost of CNTs.

This paper discusses the study of CNT reinforced MMC. The microscopic examination, EDAX and XRD analysis is carried out. Also, Hardness testing is carried out to different MMC samples to identify the best suitable combination of percentage of CNTs with the waste.

II. EXPERIMENT

A. Sample preparation

The composite material is produced by powder metallurgy method. Powder injection molding consists essentially following steps:

- i. Tailoring the powder.
- ii. Developing organic binder formulation.
- iii. Homogeneous mixing of powder and binder.
- iv. Forming parts by injection molding.
- v. Process the parts to remove organic binders.
- vi. Sintering.

B. Experimental methodology

The MMC process begins with Sigma Mixing (Mechanical Screw Mixer) in which the different elements are added at appropriate time and temperature. Initially to prepare the green sample, Mixture of steric acid and grinding waste is heated

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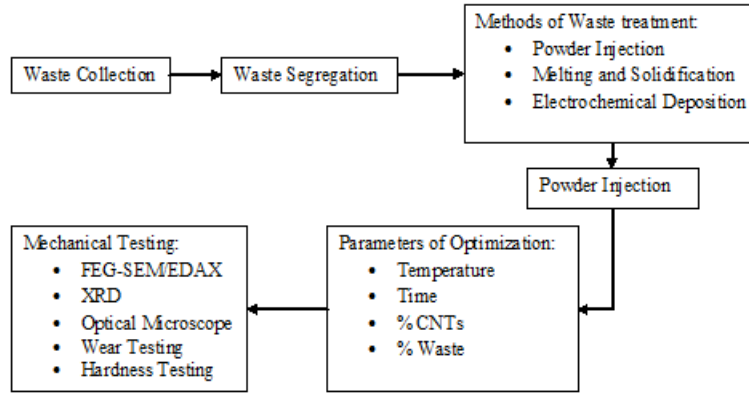


Fig. 1: Block diagram for waste treatment

Material with Binders	Sample A (86-14) (Iron 85%, CNT 01%, Binders 14%)	Sample B (88-12) (Iron 85%, CNT 03%, Binders 12%)	Sample C (90-10) (Iron 85%, CNT 05%, Binders 10%)	Sample D (91-09) (Iron 85%, CNT 06%, Binders 09%)
IRON POWDER (85%)	75 g	75 g	75 g	75 g
CNT	0.9 g (1%)	2.65 g (3%)	4.41 g (5%)	5.29 g (6%)
PARAFFIN WAX(70%)	8.65 g	7.41 g	6.18 g	5.56 g
HDPE (12%)	1.48 g	1.27 g	1.06 g	0.96 g
STEARIC ACID(18%)	2.22 g	1.91 g	1.59 g	1.43 g
Total	88.25 g	88.24 g	88.24 g	88.24 g

TABLE I: Mixing proportion of raw materials with Binders

at 60⁰C. Further wax is added at 90⁰C. Thereafter, at the same temperature, CNTs are added. Finally High - density polyethylene (HDPE) is added at 160⁰C. Further, the green mixture is used for Powder Injection Molding and various test samples are produced. Thereafter, the wax is removed by debinding process. Two types of debinding processes are carried out i.e. thermal debinding and solvent debinding. After debinding, samples are sintered between 900⁰C to 1200⁰C for 30 minutes to 120 minutes. Finally, various mechanical and physical properties are tested. The MMC flow process diagram is shown in Fig.1. For the MMC preparation, the following raw materials are used. TABLE I shows the weight percentage of each material with binders used for MMC.

III. RESULT AND DISCUSSION

1) *Solvent Debinding and Sintering Process:* The green samples are weighed and then calculated the target mass of the specimen after debinding by using the following equation:

$$M_{Target} = M_{Initial} - (0.2 * 0.7 * M_{Initial}) \quad (1)$$

The term in bracket gives the amount of wax present in the specimen. N-heptane solution is used as the solvent for wax removal. The samples are placed N-heptane bath. The bath is heated up to 60⁰C and kept at the same temperature for next 2 hours. Thereafter, the samples are removed from the bath and allowed to dry. The samples are weighed again and compared with target mass after wax removal to verify the degree of debinding (Refer Fig. 2 and TABLE II).

These brown samples then sintered (Refer Fig. 3(A) and (B)). During sintering, solid-state atomic diffusion takes place,

followed by recrystallization and grain growth. There are a significant particle movement and mass transport during sintering (Refer Fig. 3(C)).

2) *Hardness Test:* The parent iron material is Rockwell hardness about 52 *HRB*^B. TABLE III shows that as the percentage of CNTs increases, the hardness of the MMC increases. It is observed that hardness of MMC is improving by the addition of CNTs. The addition of CNTs percentage is still continued, hardness rises proportionally. However, the increase in CNTs percentage after the saturation limit, it precipitates and no further increase in hardness is observed.

3) *Microscopic analysis of samples:* Fig. 4 shows the SEM images of the Fe-CNT composite after debinding which is heated at 60⁰C. For sample A (Iron 86-14), mixing is not proper, however, after debinding, complete wax removed from the sample and material is porous. It is observed that few sights agglomerated. However, from SEM image, it is also observed that samples are properly sintered except few agglomerated regions.

4) *EDAX Test Analysis:* From TABLE IV shows the weight percentage of various materials present in the green sample. Fig. 5 shows the EDAX image of the green sample A, it has been seen that the weight percentage of parent materials after sintering are nearly same as that of green samples. The experimental analysis suggests that there is no loss of material during the sintering process. Binders are totally removed from the composite materials. Some impurities are seen in the composite. The impurities present in the parent iron powder are Si, Mg, Al, S, Na.

5) *XRD Test analysis:* From Fig. 6, XRD analysis shows the 2 peaks at 33.16, 54.05, 71.96 for sample A and 35.41,

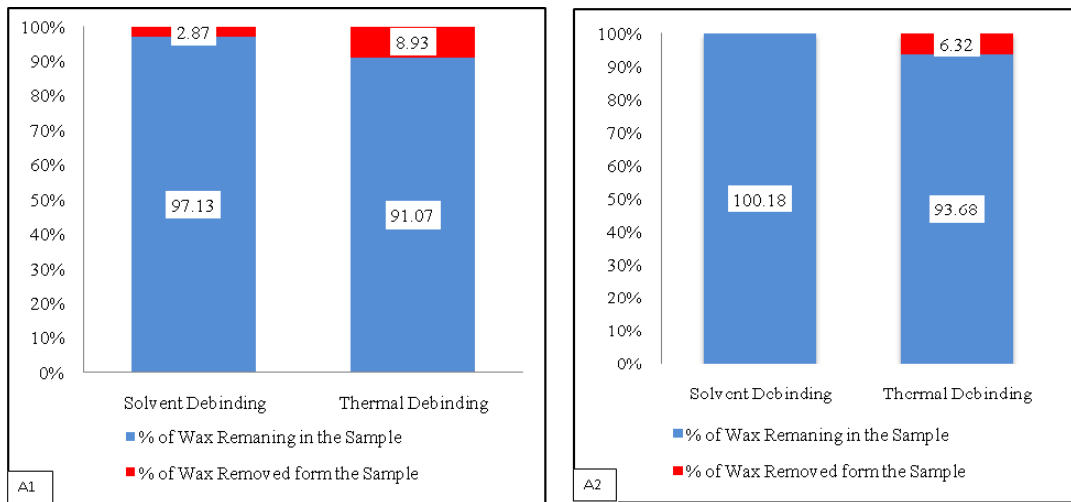


Fig. 2: Solvent and Thermal debinded Sample (A1 and A2)

Sr. No.	Sample	Initial Mass(gms)	Target Mass(gms)	Solvent Debinding		Thermal Debinding	
				Actual Mass (gms)	% Debinding	Actual Mass (gms)	% Debinding
1	A1 (86-14)	9.611	8.669	8.925	97.13	9.519	91.07
	A2 (86-14)	3.866	3.487	3.453	100.98	3.722	93.68
2	B1 (88-12)	11.541	10.574	10.678	99.03	11.361	93.07
	B2 (88-12)	4.856	4.448	4.501	98.82	4.687	94.90

TABLE II: Estimation of Solvent and Thermal debinded Sample (A and B)

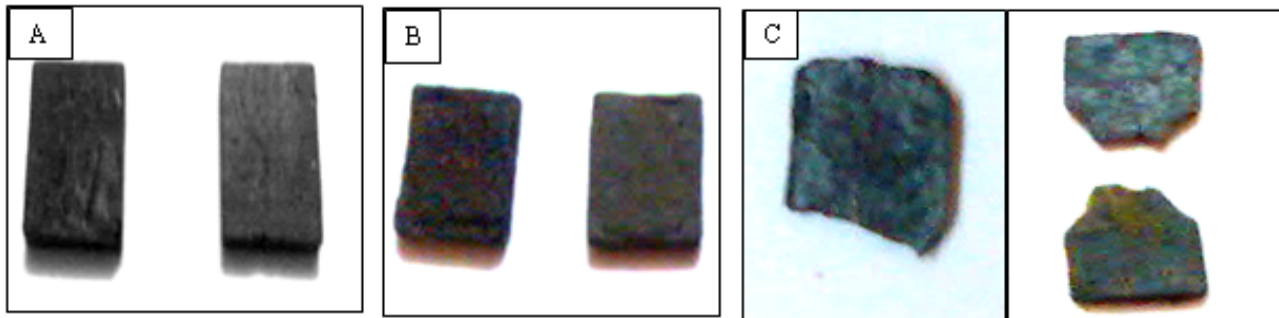


Fig. 3: (A) Green samples after PIM, (B) Brown samples after debinding and (C) Sintered Specimen

Sr. No.	Sample	Load (Kg)	Hardness Number(HRB ^B)
1	A	60	55
2	B	60	60
3	C	60	82
4	D	60	87

TABLE III: Rockwell hardness (1/16 ball indenter) for Sample A, B, C, and D

Elements Weight Percent	Sample A (86-14) (Iron 85%, CNT 01%, Binders 14%)	Sample B (88-12) (Iron 85%, CNT 03%, Binders 12%)	Sample C (90-10) (Iron 85%, CNT 05%, Binders 10%)	Sample D (91-09) (Iron 85%, CNT 06%, Binders 09%)
Fe with Oxides(Fe_3^{+} and O_2 %)	96.25	96.85	84.21	88.55
C	1.59	2.26	4.66	6.72
Si	1.54	0.57	6.40	2.51
Al	0.62	0.31	2.87	1.31
Mg	—	—	0.74	0.26
Ca	—	—	0.56	2.41
Na	—	—	0.33	0.24
S	—	—	0.22	—
Total	100	100	100	100

TABLE IV: Characterization of samples

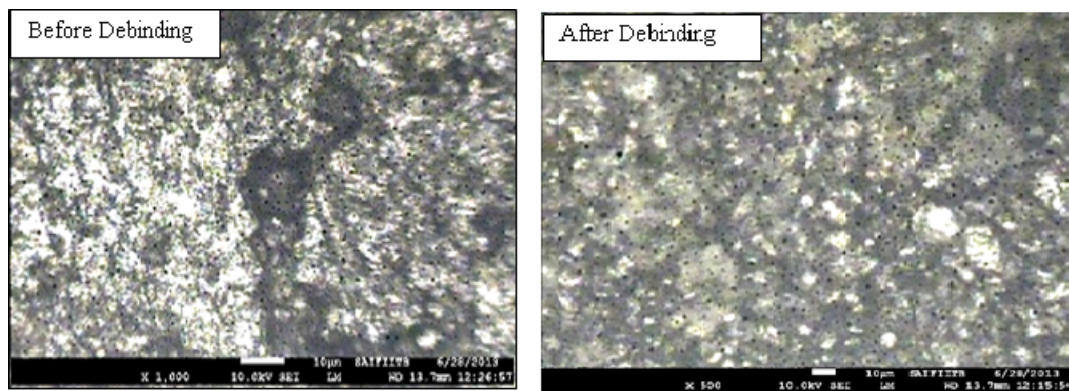


Fig. 4: SEM Image of Samples A

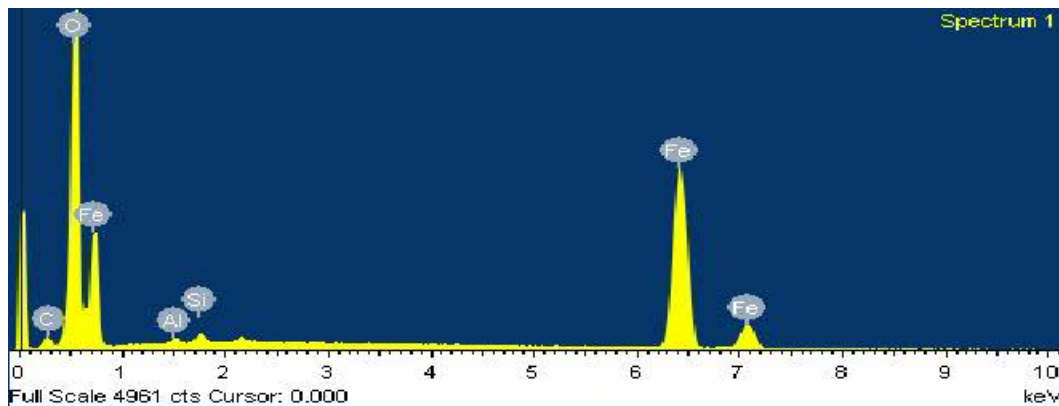


Fig. 5: EDAX Image of Sample A

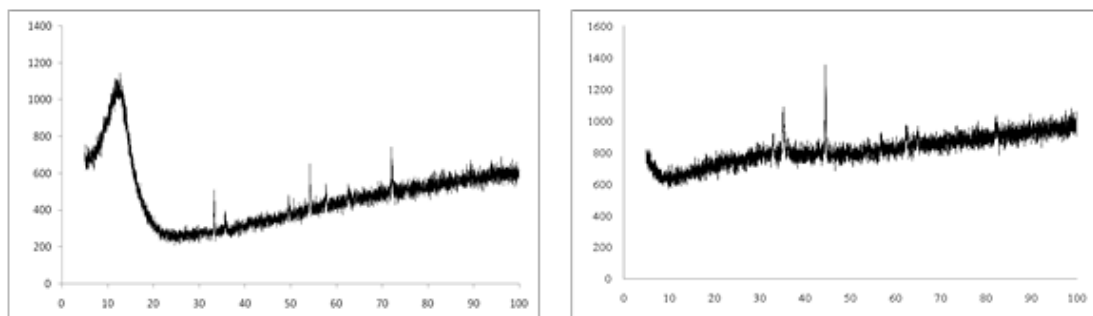


Fig. 6: XRD Image of Sample (A and B)

44.27 for sample B. It suggests that material present in the samples contains Fe in compound form. The samples produce are crystalline in nature, however, sample-B is more crystallite as compared to the sample-A. As other impurities are smaller in number, thus no significant evidence is observed in the both samples. The sample-B ordered well as compared to the sample-A, which leads to higher hardness as compared to the sample-A.

IV. CONCLUSIONS

The MMC composite has been successfully prepared and the following conclusions are drawn:

1. Sample A having 1% CNTs, Sample B having 3% CNTs,

Sample C having 5% CNTs and Sample D having 6% CNTs. It is observed that as the percentage of CNTs increase, the hardness of MMC increases. The maximum CNTs percentage to be added to MMC is 6 wt%, thereafter it precipitates out and no further improvement in hardness.

2. The debinding suggest that all wax added during processing was removed, with little distortion.

3. FEG-SEM analysis suggests that few sites agglomerates but samples possess good hardness.

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